

## FLAME-RETARDANT POLYESTER ARTIFICIAL HAIR

### TECHNICAL FIELD

The present invention relates to a flame retardant polyester based artificial hair. The hair is formed from a composition obtained by melt kneading a  
 5 polyester, a flame-retardant comprising phosphorus and/or a flame-retardant comprising bromine and at least one compound selected from the group consisting of a carbodiimide compound, a bisoxazoline compound and an isocyanate compound. More particularly, the present invention relates to a flame retardant polyester based artificial hair which has excellent spinning processability,  
 10 maintains fiber properties such as heat resistance, strength and elongation and the like, and also has excellent curl holding properties and iron setting properties.

### BACKGROUND ART

Fibers made of polyethylene terephthalate or a polyester comprising  
 15 polyethylene terephthalate as a main component have a high melting point, a high modulus of elasticity, excellent heat resistance and chemical resistance. Thus, they are widely used in curtains, carpets, clothes, blankets, sheetings, table clothes, upholstery fabrics, wall coverings, artificial hair, interior materials for automobiles, outdoor reinforcing materials, and safety nets.

20 On the other hand, human hair or artificial hair (modacrylic fibers, polyvinyl chloride fibers) or the like have conventionally been used in hair products such as wigs, hair wigs, extensions, hair bands, and doll hair. However, because it is now more difficult to obtain human hair, the use of artificial hair has

become more important. Modacrylic fibers have often been used as artificial hair materials because of their flame retardance. However, they have insufficient heat resistance.

In recent years there has been proposed, as a main component for artificial  
5 hair, a polyester typified by polyethylene terephthalate which has excellent heat resistance. However, when using a polyester as an artificial hair material, it has become necessary to add flame-retardant property for purposes of safety.

Since conventional polyester fibers are flammable, various attempts have been made to improve the flame resistance of polyester fibers. Known examples  
10 include using a fiber made of a polyester obtained by copolymerizing a flame retardant monomer containing a phosphorus atom. Examples also include adding a flame retardant to a polyester fiber.

As a method which involves copolymerizing a flame retardant monomer, for example, several methods have been proposed. Copolymerizing a phosphorus  
15 compound with excellent heat stability having a phosphorus atom as a ring member is disclosed in Japanese Patent Publication No. 55-41610.

Copolymerizing carboxyphosphinic acid is also disclosed in Japanese Patent Publication No. 53-13479. Also, copolymerizing or adding a phosphorus compound to a polyester containing a polyarylate is disclosed in Japanese Laid-  
20 open Patent No.11-124732.

As artificial hair to which flame retardant technology is applied, a polyester fiber copolymerized with a phosphorus compound has been proposed in Japanese Laid-open Patent No.03-27105, for example. However, since artificial hair is

required to highly flame resistant, the copolymer polyester fiber used for artificial hair must have a high copolymerization amount. This results in a significant decrease in heat resistance of the polyester, and causes other problems which make it difficult to perform melt spinning. Also, when a flame approaches, the  
5 artificial hair does not catch fire and burn, but becomes molten and drips.

On the other hand, as a method which involves adding a flame retardant, it has been proposed to add a halogenated cycloalkane compound as fine particles to a polyester fiber, as disclosed in Japanese Patent Publication No. 03-57990, or add a bromine-containing alkylcyclohexane to a polyester fiber, as disclosed in  
10 Japanese Patent Publication No. 01-24913. However, in the method which involves adding a flame retardant to a polyester fiber, in order to achieve sufficient flame retardance the addition treatment temperature must be as high as 150°C or more, the addition treatment time must be long, or a large amount of a flame retardant must be used. This causes problems such as deteriorated fiber properties,  
15 reduced productivity and an increased production cost.

On the other hand, recently it has become necessary to collect and recycle disposed polyester products in the interest of reuse of material and environmental conservation. For example, bottles for liquid foods made of polyethylene terephthalate use polyester in a large amount and consumption of those bottles is  
20 expected to increase in the future. Accordingly it is worthwhile to collect and recycle those polyethylene terephthalate bottles and, in fact, all attempts to do so have been launched.

In a conventional process for producing polyester fibers for clothes, conditions for drawing are adjusted to produce polyester fibers having an elongation at break of 30%. When the recycled polyester is spun to produce fibers having such conditions for drawing, thread breakages frequently take place due to additives and foreign bodies in the polyester. This results in substantially deteriorated operational efficiency. Accordingly, it has been difficult to spin recycled polyester to produce versatile multi-filaments for clothes.

In order to commercially produce polyester fibers while maintaining fiber properties, polyester fibers must contain impurities of 5% or less. Therefore, recycled polyester bottles separated from impurities must be used as raw materials. However, since bottles for liquid food products are filled with heat sterilized liquid, the bottle's mouth part is required to be heat resistant to a certain extent. Therefore materials other than polyester are often used for the mouth part of the polyester bottles. When polyester bottles are used as raw materials, it is difficult to remove these different materials..

As described above, artificial hair fiber which retains the fiber properties possessed by conventional polyester fiber; such as heat resistance, strength and elongation and the like, and also has excellent setting properties and flame retardance, has not yet been produced. Furthermore, artificial hair fiber formed from collected and recycled polyester which has excellent setting properties and flame retardance has not yet been produced.

## DISCLOSURE OF THE INVENTION

An object of the present invention is to provide a flame retardant polyester based artificial hair which has excellent spinning processability, maintains fiber properties such as heat resistance, strength and elongation and the like and also has  
5 excellent curl holding properties and iron setting properties, is achieved by solving the aforescribed existing problems.

In order to solve the aforescribed problems, the inventors have conducted intensive studies. They have found that a flame retardant polyester based artificial hair which have excellent spinning processability, maintains fiber properties such  
10 as heat resistance, strength and elongation and the like and also has excellent curl holding properties and iron setting properties, can be obtained by melt spinning a composition comprising a polyester, a phosphorus containing flame retardant and/or a bromine containing flame retardant and at least one compound selected from the group consisting of a carbodiimide compound, a bisoxazoline compound  
15 and an isocyanate compound.

Specifically, the present invention relates to a flame retardant polyester based artificial hair, formed from a composition having intrinsic viscosity of 0.5 to 1.4 which is obtained by melt kneading 100 parts by weight of (A) a polyester made of one or more of polyalkylene terephthalate or a copolymer polyester  
20 comprising polyalkylene terephthalate as a main component, 5 to 30 parts by weight of (B) a phosphorus containing flame retardant and/or a bromine containing flame retardant and 0.05 to 10 parts by weight of (C) at least one

compound selected from the group consisting of a carbodiimide compound, a bisoxazoline compound and an isocyanate compound.

Furthermore, the present invention relates to the flame retardant polyester based artificial hair wherein the fiber is preferably non-crimped and spun dyed,  
5 and the size of a single fiber is 10 to 100 dtex.

According to the present invention, flame retardant polyester based artificial hair can be obtained which has excellent spinning processability, maintains fiber properties such as heat resistance, strength and elongation and the like and also has excellent curl holding properties and iron setting properties.

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#### BEST MODE FOR CARRYING OUT THE INVENTION

The flame retardant polyester based artificial hair of the present invention is a fiber obtained by melt spinning a composition having an intrinsic viscosity of 0.5 to 1.4, which composition is obtained by melt kneading (A) a polyester made of  
15 one or more of polyalkylene terephthalate or a copolymer polyester comprising polyalkylene terephthalate as a main component, (B) a phosphorus containing flame retardant and/or a bromine containing flame retardant and (C) at least one compound selected from the group consisting of a carbodiimide compound, a bisoxazoline compound and an isocyanate compound.

20 Examples of the polyalkylene terephthalate or the copolymer polyester comprising polyalkylene terephthalate as a main component, which is contained in the polyester (A) used in the present invention, include polyalkylene terephthalates such as polyethylene terephthalate, polypropylene terephthalate, and polybutylene

terephthalate, and a copolymer polyester comprising such polyalkylene terephthalate as a main component and a small amount of a copolymerization component. Further flakes of disposed PET bottles can also be reused. The phrase "comprising as a main component" refers to "comprising in an amount of 5 80 mol% or more".

Examples of the copolymerization component include: polycarboxylic acids such as isophthalic acid, orthophthalic acid, naphthalenedicarboxylic acid, paraphenylenedicarboxylic acid, trimellitic acid, pyromellitic acid, succinic acid, glutaric acid, adipic acid, suberic acid, azelaic acid, sebacic acid, and 10 dodecanedioic acid, and their derivatives; dicarboxylic acids having sulfonic acid salt such as 5-sodium sulfoisophthalic acid, dihydroxyethyl 5-sodium sulfoisophthalate, and their derivatives; 1,2-propanediol, 1,3-propanediol, 1,4-butanediol, 1,6-hexanediol, neopentyl glycol, 1,4-cyclohexanedimethanol, diethylene glycol, polyethylene glycol, trimethylolpropane, pentaerythritol, 4- 15 hydroxybenzoic acid, and  $\epsilon$ -caprolactone.

Typically, the copolymer polyester is preferably produced by adding a small amount of a copolymerization component to a main component which is a polymer of terephthalic acid and/or its derivative (for example, methyl terephthalate) and alkylene glycol, and reacting these components with a view 20 toward stability and convenience for handling. However, the copolymer polyester may be produced by adding a small amount of a monomer or oligomer component as a copolymerization component to a main component which is a mixture of

terephthalic acid and/or its derivative (for example, methyl terephthalate) and alkylene glycol, and polymerizing the components.

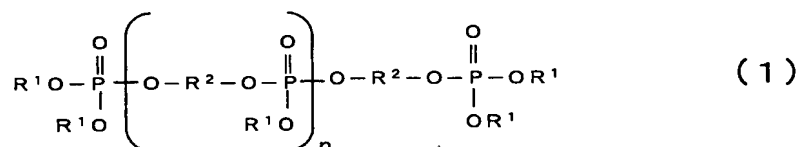
The copolymer polyester may be any copolymer polyester in which the copolymerization component is polycondensed with the main chain and/or the side  
5 chain of polyalkylene terephthalate as a main component. There are no particular limitations to the manner of copolymerization and the like.

Examples of the copolymer polyester comprising polyalkylene terephthalate as a main component include: a polyester obtained by copolymerizing polyethylene terephthalate as a main component with ethylene glycol ether of  
10 bisphenol A; a polyester obtained by copolymerizing polyethylene terephthalate as a main component with 1,4-cyclohexanedimethanol; and a polyester obtained by copolymerizing polyethylene terephthalate as a main component with dihydroxyethyl 5-sodium sulfoisophthalate. The polyalkylene terephthalate and its copolymer polyester may be used singly or in a combination of two or more.

15 Preferable examples thereof include polyethylene terephthalate, polypropylene terephthalate, polybutylene terephthalate, and a copolymer polyester (a polyester obtained by copolymerizing polyethylene terephthalate as a main component with ethylene glycol ether of bisphenol A; a polyester obtained by copolymerizing polyethylene terephthalate as a main component with 1,4-  
20 cyclohexanedimethanol; a polyester obtained by copolymerizing polyethylene terephthalate as a main component with dihydroxyethyl 5-sodium sulfoisophthalate; or the like). A mixture of two or more of these is also preferable.

The phosphorus containing flame retardant and/or a bromine containing flame retardant (B) used in the present invention is not particularly limited, and generally used phosphorus containing flame retardants can be used.

Examples of the phosphorus containing flame retardant used for the phosphorus containing flame retardant and/or the bromine containing flame retardant (B) used in the present invention include a phosphate compound, a phosphonate compound, a phosphinate compound, a phosphine oxide compound, a phosphonite compound, a phosphinite compound, a phosphine compound and a condensed phosphate compound represented by the general formula (1).



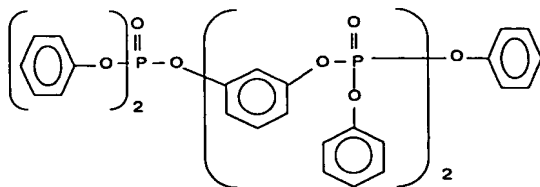
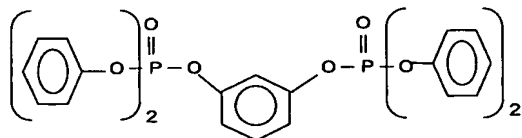
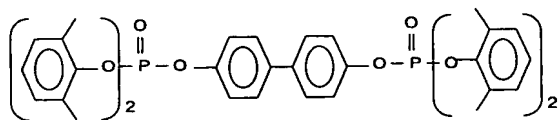
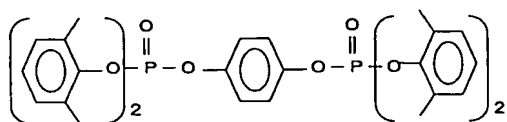
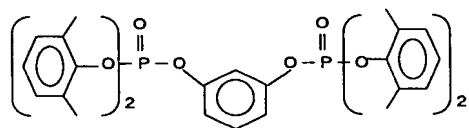
wherein R<sup>1</sup> groups represent a monovalent aromatic hydrocarbon group or aliphatic hydrocarbon group, and may be the same or different; R<sup>2</sup> represents a divalent aromatic hydrocarbon group, and when two or more R<sup>2</sup> groups are present, the groups may be the same or different; and n is 0 to 15.

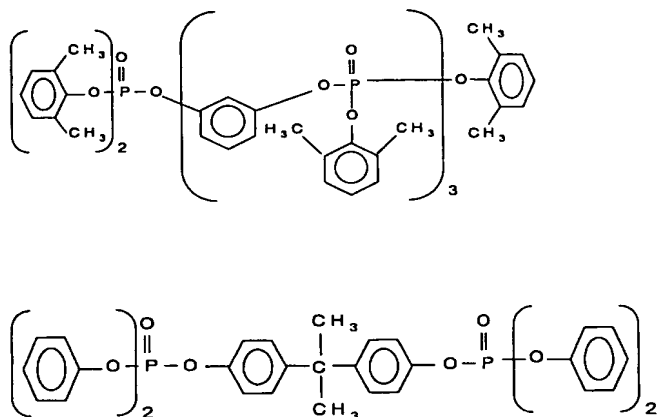
These may be used alone or as a mixture of two or more thereof.

Specific examples of the phosphorus containing flame retardant include trimethyl phosphate, triethyl phosphate, tributyl phosphate, tri(2-ethylhexyl) phosphate, triphenyl phosphate, tricresyl phosphate, trixylenyl phosphate, tris(isopropylphenyl) phosphate, tris(phenylphenyl) phosphate, trinephthyl phosphate, cresylphenyl phosphate, xylenyl diphenyl phosphate, triphenyl

phosphine oxide, tricresyl phosphine oxide, diphenyl methanephosphonate and diethyl phenylphosphonate, as well as resorcinol polyphenyl phosphate, resorcinol poly(di-2,6-xylyl) phosphate, bisphenol A polycresyl phosphate, hydroquinone poly(2,6-xylyl) phosphate, and condensed phosphate compounds represented by

5 the following formulas:





Among these compounds, the condensed phosphate compounds represented by the general formula (1) are preferable.

5        The bromine containing flame retardant used for the phosphorus containing flame retardant and/or the bromine containing flame retardant (B) used in the present invention is not particularly limited, and generally used bromine containing flame retardants can be employed.

Specific examples of the bromine containing flame retardant used for the  
 10    phosphorus containing flame retardant and/or the bromine containing flame retardant (B) used in the present invention include pentabromotoluene, hexabromobenzene, decabromobiphenyl, decabromodiphenyl ether, bis(tribromophenoxy)ethane, tetrabromophthalic anhydride, ethylene bis(tetrabromophthalimide), ethylene bis(pentabromophenyl),  
 15    octabromotrimethylphenylindane, bromine-containing phosphate esters such as tris(tribromoneopentyl)phosphate, brominated polystyrenes, brominated polybenzyl acrylates, brominated epoxy oligomers, brominated phenoxy resins, brominated polycarbonate oligomers, derivatives of tetrabromobisphenolA such as

tetrabromobisphenolA, tetrabromobisphenolA-bis (2, 3-dibromopropyl ether),  
tetrabromobisphenolA-bis (allylether), tetrabromobisphenolA-bis(hydroxyethyl  
ether), bromine-containing triazine based compounds such as  
tris(tribromophenoxy) triazine and bromine-containing isocyanuric acid based  
5 compound such as tris(2, 3-dibromopropyl) isocyanurate. These may be used  
alone or at least two kinds thereof may be used in combination.

Among these, a bromine-containing phosphate ester based flame retardant,  
a brominated polystyrene based flame retardant, a brominated benzyl acrylate  
based flame retardant, a brominated polycarbonate based flame retardant, a  
10 derivative of tetrabromobis phenol-A, a bromine-containing triazine based  
compound and a bromine-containing isocyanuric acid based compound are  
preferable.

The amount of the phosphorus containing flame retardant and/or the  
bromine containing flame retardant (B) is preferably 5 to 30 parts by weight, more  
15 preferably 6 to 25 parts by weight, and, further more preferably 7 to 20 parts by  
weight based on 100 parts by weight of the polyester (A). If the amount of the  
phosphorus containing flame retardant and/or the bromine containing flame  
retardant (B) is less than 5 parts by weight, the effect of flame resistance tends to  
hardly be obtained, and if more than 30 parts by weight, mechanical properties,  
20 heat resistance and drip resistance tend to be damaged.

The carbodiimide compound used as (C) component in the present  
invention is a compound having at least two carbodiimide groups represented by (-  
N=C=N-) and is not particularly limited. Examples of carbodiimide compound

include mono- or di-carbodiimide compounds such as diphenyl carbodiimide, dicyclohexyl carbodiimide, di-2,6-dimethylphenyl carbodiimide, diisopropyl carbodiimide, dioctyldecyl carbodiimide, di-o-tolylcarbodiimide, N-tolyl-N'-phenyl carbodiimide, N-tolyl-N'-cyclohexyl carbodiimide, di-p-tolylcarbodiimide, 5 di-p-nitrophenyl carbodiimide, di-p-aminophenyl carbodiimide, di-p-hydroxyphenyl carbodiimide, di-p-chlorophenyl carbodiimide, di-o-chlorophenyl carbodiimide, di-3,4-dichlorophenyl carbodiimide, di-2,5-dichlorophenyl carbodiimide, p-phenylene-bis-o-tolylcarbodiimide, p-phenylene-bis-dicyclohexyl carbodiimide, p-phenylene-bis-di-p-chlorophenyl carbodiimide, hexamethylene- 10 bis-di-cyclohexyl carbodiimide, ethylene-bis-diphenyl carbodiimide and ethylene-bis-di-cyclohexyl carbodiimide; polycarbodiimides such as poly(1,6-hexamethylene carbodiimide), poly(4,4'-methylene-bis-cyclohexyl carbodiimide), poly(1,3-cyclohexylene carbodiimide, and poly(1,4-cyclohexylene carbodiimide); and aromatic polycarbodiimides such as poly(4,4'-diphenylmethane carbodiimide), 15 poly(3,3'-dimethyl-4,4'-diphenylmethane carbodiimide), poly(naphtylene carbodiimide), poly(p-phenylene carbodiimide), poly(m-phenylene carbodiimide), poly(tolylcarbodiimide), poly(diisopropylphenylene carbodiimide), poly(methyl-diisopropylphenylene carbodiimide), poly(triethylphenylene carbodiimide) and poly(triisopropylphenylene carbodiimide). As commercial available products, 20 there are Bayer AG's STABAXOL I and STABAXOL P (both registered trade names). The aforementioned carbodiimide compounds may be used either alone or in combination of two or more.

The bisoxazoline compound used as component (C) in the present invention is not particularly limited. Examples of the oxazoline compound include 2,2'-methylene-bis(2-oxazoline), 2,2'-ethylene-bis(2-oxazoline), 2,2'-ethylene-bis (4-methyl-2-oxazoline), 2,2'-propylene-bis(2-oxazoline), 2,2'-tetramethylene-bis(2-oxazoline), 2,2'-hexamethylene-bis(2-oxazoline), 2,2'-octamethylene-bis(2-oxazoline), 2,2'-p-phenylene-bis(2-oxazoline), 2,2'-p-phenylene-bis(4-methyl-2-oxazoline), 2,2'-p-phenylene-bis(4,4'-dimethyl-2-oxazoline), 2,2'-p-phenylene-bis(4-phenyl-2-oxazoline), 2,2'-m-phenylene-bis(2-oxazoline), 2,2'-m-phenylene-bis(4-methyl-2-oxazoline), 2,2'-m-phenylene-bis(4,4'-dimethyl-2-oxazoline), 2,2'-m-phenylene-bis(4-phenyl-2-oxazoline), 2,2'-o-phenylene-bis(2-oxazoline), 2,2'-bis(2-oxazoline), 2,2'-bis(4-methyl-2-oxazoline), 2,2'-bis(4-phenyl-2-oxazoline), and the like. The aforementioned bisoxazoline compounds may be used either alone or in combination of two or more.

The isocyanate compound used as component (C) in the present invention may be various aliphatic diisocyanates or aromatic diisocyanates. The alkylene group in the aliphatic diisocyanates is not limited to a straight structure but may be branched structure or alicyclic structure. The alkylene group may contain oxygen atom. The aromatic diisocyanates is not particularly limited provided that divalent aromatic hydrocarbon group is contained in the molecule.

Examples of aliphatic diisocyanates include 1,3-trimethylene diisocyanate, 1,4-tetramethylene diisocyanate, 1,6-hexamethylene diisocyanate, 1,9-nonamethylene diisocyanate, 1,10-decamethylene diisocyanate, 1,12-dodecamethylene diisocyanate, hexamethylene diisocyanate-biuret, 2,2,4-

trimethylhexamethylene diisocyanate, 2,4,4-trimethylhexamethylene diisocyanate, isophorone diisocyanate, 1,4-cyclohexane diisocyanate, 4,4'-dicyclohexylmethane diisocyanate, hydrogenated xylylene diisocyanate, 2,2'-diethylether diisocyanate and the like.

- 5           Examples of aromatic diisocyanetes include p-phenylene diisocyanate, tolylene diisocyanate, xylylene diisocyanate, 4,4'-diphenyl diisocyanate, 1,5-naphthalene diisocyanate, 4,4'-diphenylmethane diisocyanate, 3,3'-methyleneditolylene-4,4'-diisocyanate, tolylenediisocyanate-trimethylolpropane adduct, triphenylmethane triisocyanate, 4,4'-diphenylether diisocyanate,
- 10   tetrachlorophenylene diisocyanate, 3,3'-dichloro-4,4'-diphenylmethane diisocyanate and triisocyanate phenylthiophosphate. The aforementioned isocyanate compounds may be used either alone or in combination of two or more.

- The amount of the at least one compound selected from the group consisting of a carbodiimide compound, a bisoxazoline compound and an isocyanate
- 15   compound (C) to be used in the present invention is 0.05-10 parts by weight, preferably 0.1-8 parts by weight, more preferably 0.2-6 parts by weight based on 100 parts by weight of the polyester (A). If the amount of the at least one compound selected from the group consisting of a carbodiimide compound, a bisoxazoline compound and an isocyanate compound (C) to be used is less than
- 20   0.05 parts by weight, the advantageous effects that the flowability is lowered and bleeding is prevented tend to be insufficient. If it exceeds 10 parts by weight, flame retardance and mechanical strength of the flame retardant polyester based artificial hair tend to be deteriorated.

The composition of the present invention obtained by melt kneading the components (A), (B) and (C) can have a excellent spinning property by controlling the amount of component (C) and adjusting the intrinsic viscosity to 0.5 to 1.4. The composition has an intrinsic viscosity of preferably 0.5 to 1.4, and more  
5 preferably 0.6 to 1.2. If the intrinsic viscosity is less than 0.5, the resulting fiber tends to have reduced mechanical strength. If more than 1.4, the melt viscosity is increased as the molecular weight is increased, and thus the fiber tends to be melt spun only with difficulty, and to have a non-uniform size.

The flame retardant polyester composition used in the present invention can  
10 be produced by, for example, dry blending the components (A), (B) and (C) in advance and then melt kneading the components in various conventional kneading machines. Examples of the kneading machines include a single-screw extruder, twin-screw extruder, roll, Banbury mixer and kneader. Of these, a twin-screw extruder is preferable in terms of adjustment of the kneading degree and  
15 convenience of operation.

The composition for the present invention can be obtained by, for example, melt-kneading at a barrel temperature of 260 to 300°C, a discharge mount of 50 to 150 kg/hr, and a screw rotational speed of 150 to 200 rpm with a twin screw extruder having a screw diameter of 45 mm, taking up a strand out of dies, and  
20 after cooling with water, pelletizing the strand with a strand cutter.

The flame retardant polyester based artificial hair of the present invention can be produced by melt spinning the above-described polyester composition by a typical melt spinning process. Specifically, a spun yarn can be obtained by, for

example: melt spinning the composition while setting an extruder, gear pump, spinneret, and the like at a temperature of 270 to 310°C; allowing the spun yarn to pass through a heat sleeve; then cooling the yarn to a glass transition temperature or lower; and taking off the yarn at a rate of 50 to 5,000 m/min. The size of the spun yarn can also be controlled by cooling the yarn in a tank filled with cooling water. The temperature or length of the heat sleeve, the temperature or spraying amount of cooling air, the temperature of the cooling tank, the cooling time, and the take-off rate can be appropriately adjusted according to the discharge amount and the number of holes in the spinneret.

10       The resulting spun yarn may be hot drawn by either a two-step process comprising winding up the spun yarn once and then drawing the yarn, or a direct spinning and drawing process comprising successively drawing the spun yarn without winding. Hot drawing is carried out by a one-stage drawing process or a multistage drawing process. As a heating means in hot drawing, a heat roller, heat plate, steam jet apparatus, hot water tank, or the like can be used. These can be  
15       appropriately used in combination.

      The polyester based artificial hair of the present invention may contain various additives such as a heat resistant agent, a photostabilizer, a fluorescent agent, an antioxidant, an antistatic agent, a pigment, a plasticizer, and a lubricant  
20       as required. The fiber containing a pigment can be provided as a spun dyed fiber.

      When the polyester based artificial hair of the present invention thus obtained is a fiber in the form of a non-crimped fiber, and has a size of usually 10 to 100 dtex, and furthermore 20 to 90 dtex, it is suitable for artificial hair.

Preferably, the artificial hair has heat resistance sufficient to allow a thermal beauty appliance (hair iron) to be used thereon at 160 to 200°C. Preferably, the artificial hair catches fire only with difficulty, and has self-extinguishing properties.

5           When the polyester based artificial hair of the present invention is spun dyed, the artificial hair can be used as is. When the artificial hair is not spun dyed, it can be dyed under the same conditions as with a common flame retardant polyester fiber. The pigment, dye, adjuvant, or the like used for dyeing preferably exhibits excellent weather resistance and flame retardance.

10           The polyester based artificial hair of the present invention exhibits excellent curl setting properties when a thermal beauty appliance (hair iron) is used, and also exhibits excellent curl holding properties. The artificial hair can be appropriately matted with irregularities on the fiber surface and can be used for artificial hair. Further, an oil agent such as a fiber surface treating agent or a  
15           softening agent can provide the fiber with feeling and texture closer to human hair.

          The flame retardant polyester based artificial hair of the present invention may be used in combination with another material for artificial hair, such as a modacrylic fiber, a polyvinyl chloride fiber, or a nylon fiber, or it may be used in combination with human hair.

20           Next, the present invention will be described in more detail with reference to Examples. However, it should be understood that the present invention is not limited thereto.

## EXAMPLES

Properties values in the present invention are measured as follows.

### (Intrinsic viscosity of polyester)

A solution at a concentration of 0.5 g/dl in a mixed solvent of equal weights  
5 of phenol and tetrachloroethane is measured for its relative viscosity at 25°C with  
an Ubbelohde viscometer, and the intrinsic viscosity is calculated from the  
following formula:

$$\begin{aligned} [\eta] &= \lim_{C \rightarrow 0} \eta_{sp} / C = \lim_{C \rightarrow 0} (\eta_{rel} - 1) / C \\ &= \lim_{C \rightarrow 0} (\eta - \eta_0) / \eta_0 C \end{aligned}$$

wherein  $\eta$  is the viscosity of the solution,  $\eta_0$  is the viscosity of the solvent,  
10  $\eta_{rel}$  is relative viscosity,  $\eta_{sp}$  is specific viscosity,  $[\eta]$  is intrinsic viscosity, and C is  
the concentration of the solution.

### (Strength and elongation)

Tensile strength and elongation of a filament are measured using INTESCO  
Model 201 manufactured by INTESCO Co., Ltd. Both 10 mm-long ends of one  
15 40 mm-long filament are sandwiched in a board (thin paper) to which a two-sided  
tape applied with an adhesive is bonded, and are air-dried overnight to prepare a  
sample with a length of 20 mm. The sample is mounted on a test machine, and a  
test is carried out at a temperature of 24°C, at a humidity of 80% or less, at a load  
of 1/30 gF × size (denier) and at a tensile rate of 20 mm/min to measure strength

and elongation. The test is repeated ten times under the same conditions, and the average values are defined as strength and elongation of the filament.

**(Heat resistance)**

A thermal shrinkage ratio of filaments is used as an index of heat resistance.

- 5 A thermal shrinkage ratio is measured by using a SC5200H thermal analyzer TMA/SS150C manufactured by Seiko Instruments Inc. Ten pieces of filaments having a length of 10 mm are adopted, and a load of 5.55 mg/dtex is applied thereto. The thermal shrinkage ratio of the filaments is measured at a speed of temperature increase of 3°C/min in a range of 30 to 280°C.

10 **(Flame retardance)**

- A filament having a size of 50 dtex is cut into filaments with a length of 150 mm each. Filaments with a weight of 0.7 g are bundled, with one end of the bundle sandwiched by a clamp, and the bundle is fixed on a stand and hung vertically. The fixed filaments with an effective length of 120 mm are brought  
15 into contact with a 20 mm-long flame for 3 seconds and burned.

Flammability is evaluated as follows.

Very good: After-flame time is 0 second (Filaments do not catch fire)

Good: After-flame time is less than 3 seconds

Fair: After-flame time is 3 to 10 seconds

- 20 Bad: After-flame time is more than 10 seconds

Drip resistance is evaluated as follows.

Very good: The number of drips is 0

Good: The number of drips is 5 or less

Fair: The number of drips is 6 to 10

Bad: The number of drips is 11 or more

**(Curl setting properties)**

Straw-haired filaments are wound around a pipe with a diameter of 32 mm.

- 5 Curl setting is performed by steam under conditions at 120°C and relative humidity of 100% for 60 minutes, and aging is performed at room temperature for 60 minutes. Then, one end of the curled filaments is fixed, and the filaments are hung down to visually evaluate curling state. Curling state is regarded as an indication of the ease of curling. A state with a smaller length and with curl in
- 10 sufficient form is preferable.

Good: curl in sufficient form

Fair: curl relaxed a little

Bad: curl loosened and form collapsed

**(Iron setting properties)**

- 15 Iron setting properties are an index of the extent to which a hair iron can perform curl setting easily and hold the curl shape. Filaments are loosely sandwiched in a hair iron heated to 180°C, and pre-heated three times by rubbing. Adhesion among the filaments, combing, crimping and end breakage of the filaments are visually evaluated at this time. Next, the pre-heated filaments are
- 20 wound around the hair iron and held for 10 seconds, after which the iron is withdrawn. The degree of ease with which the iron is withdrawn (rod withdrawal properties), and curl holding properties when the iron is withdrawn are visually evaluated.

# **EXAMPLES (No. 1 to 8)**

Compositions having components ratios shown in Table 1 below were dried so as to have a moisture content of 100 ppm or less. To each of these compositions, 2 parts of a coloring polyester pellet PESM6100 BLACK (manufactured by Dainichiseika Color & Chemicals Mfg. Co., Ltd., carbon black content: 30%, polyester contained in the component (A)) were added. The compositions were dry blended. The blend was fed into a twin-screw extruder and melt kneaded at 280°C to form a pellet. Then, the pellet was dried to a moisture content of 100 ppm or less. Next, the pellet was put into a melt spinning machine, and the molten polymer was discharged from a spinneret having round cross-sectional nozzle holes with a nozzle diameter of 0.5 mm and each nozzle at 280°C. The discharged polymer was air cooled with cooling wind of 20°C and rolled up at a rate of 100 m/min to obtain un-drawn yarns. The obtained un-drawn yarn was drawn to form a four-fold drawn yarn by using a heat roll heated at 85°C, heat treated by using a heat roll heated at 200°C and rolled up at a speed of 30 m/min to obtain polyester based fibers (multi-filament) with a size of around 50 dtex.

**Table 1**

		Example								Comparative Example	
		1	2	3	4	5	6	7	8	1	2
(A)	EFG-10 *1	100	100	100	100	100	100			100	
	collected PET flake *2							100	100		100
(B)	PX-202 *3	15	15	15				15		15	15
	SR-T5000 *4				12	12	12		12		
(C)	Stabaxol P *5	4			2				4		



properties (180°C)	Shrinkage/thread breakage	Good	Good	Good	Good	Good	Good	Good	Good
	Rod withdrawal	Good	Good	Fair	Good	Good	Good	Good	Good
	Curl holding	Good	Good	Good	Good	Good	Good	Good	Good

### (Comparative Example 1 and 2)

The compositions having component ratios shown in Table 1 were dried so as to have a moisture content of 100ppm or less and polyester fibers (multi-  
5 filament) having a single fiber size of around 50 dtex were obtained in the same manner as Examples.

Strength and elongation, thermal shrinkage ratio, flame retardance, curl holding properties, iron setting properties of the resulting fiber were evaluated.

The results are shown in Table 3 below.

10

Table 3

		Comparative Example	
		1	2
Intrinsic viscosity of composition (IV)		0.73	0.73
Spinning processability		Fair	Bad
Size (dtex)		49	48
Strength (cN/dtex)		1.8	1.5
Elongation (%)		69	72
Thermal shrinkage ratio at 180°C (%)		4.1	4.6
Frame retardance	Flammability	Fair	Fair
	Drip resistance	Bad	Bad
Curl setting properties		Fair	Fair
Iron setting properties (180°C)	Adhesion	Good	Good
	Shrinkage/thread breakage	Good	Good

	Rod withdrawal	Bad	Bad
	Curl holding	Fair	Fair

As shown in Tables 2 and 3, excellent heat resistance, curl holding properties and iron setting properties were observed in Examples when compared to Comparative Examples. It is also observed that the intrinsic viscosity can be controlled resulting in excellent spinning processability by using a carbodiimide compound, a bis-oxazoline compound or an isocyanate compound.

Accordingly, it was confirmed that the present artificial hair using a carbodiimide compound, a bis-oxazoline compound or an isocyanate compound can be utilized effectively for an artificial hair which maintains mechanical and thermal properties of polyester and has improved heat resistance and iron setting properties and the like, compared with conventional artificial hair.

#### INDUSTRIAL APPLICABILITY

According to the present invention, it is possible to provide a flame retardant polyester based artificial hair which has an excellent spinning processability, curl holding properties and iron setting properties while maintaining fiber properties such as heat resistance, strength and elongation and the like.